



1 242 309

(11) (A) No.

(45) ISSUED 880927

(52) CLASS 23-362
C.R. CL. 202-92

(51) INT. CL. B01D 3/00⁴

(19) (CA) **CANADIAN PATENT** (12)

(54) Carrying Out Chemical Reactions and Simultaneously
Separating a Product Mixture Into Several Fractions
by Means of a Distillation Column

(72) Kaibel, Gerd,
Germany (Federal Republic of)

(73) Granted to BASF Aktiengesellschaft
Germany (Federal Republic of)

(21) APPLICATION No. 452,048

(22) FILED 840416

(30) PRIORITY DATE Germany (Federal Republic of)
(P 33 14 395.1) 830421

No. OF CLAIMS 4

Canada

DISTRIBUTED BY THE PATENT OFFICE, OTTAWA
CCA-274 (11-82)

BEST AVAILABLE COPY

452048

Abstract of the Disclosure: A chemical reaction is carried out and at the same time a product mixture is separated into several fractions by means of a distillation column which, in parts, is divided into a reaction section and a distillation section by separating units which are effective in the longitudinal direction and completely or partly prevent cross-mixing of liquid and/or vapor streams, by a method in which one or more reactants and, where relevant, a catalyst are fed into the reaction section, and at the same time one or more medium-boiling fractions, which can consist of reactants and/or reaction products and are free, or substantially free, from contamination by top and bottom fractions, are taken off in vapor or liquid form from the distillation section.

Carrying out chemical reactions and simultaneously separating a product mixture into several fractions by means of a distillation column

The present invention relates to a method of carrying out chemical reactions and simultaneously separating a product mixture into several fractions by means of a distillation column which, in parts, is divided into a reaction section and a distillation section by separating units which are effective in the longitudinal direction and completely or partly prevent cross-mixing of liquid and/or vapor streams.

Carrying out chemical reactions in distillation columns so that the reaction and separation of substances by distillation are overlapping steps taking place side by side in the same space is a procedure which is well established in practice. Frequently, this procedure has advantages with regard to energy requirement and capital costs in comparison with procedures in which the reaction and the separation by distillation take place in separate process steps, ie. the reaction taking place in a reactor and the separation of substances taking place in a downstream distillation column. Examples of chemical reactions which are advantageously carried out in distillation columns are esterifications, transesterifications, hydrolyses, acetalization and acetal cleavage reactions. Particularly suitable distillation columns are those used for equilibrium reactions in which all of the reactants have similar boiling points while the reaction products boil at substantially higher and/or lower temperatures. However, even in the case of reactions where this optimum boiling behavior is not exhibited, distillation columns can be reasonably employed since they permit the advantageous counter-current flow of the reactants to be realized. This results in higher conversions than in the case of direct mixing without counter-current flow.

For example, in a reaction $A+B \rightleftharpoons$

C+D (reaction products), where the boiling sequence in order of increasing boiling points is A, C, B, D and reactant A is introduced in excess instead of in a stoichiometric amount, a mixture of A and C can be taken off from the distillation column as a top product, while D can be taken off as a bottom product. A further, downstream distillation column is necessary for separating the top products A and C. The same applies when one reactant is the highest boiling component. In this case too, and for the same boiling sequence as above, an additional distillation column is required in order to obtain the unreacted reactant B and the reaction product D in pure form. Distillation columns are used even where the reaction products are neither the lowest boiling nor the highest boiling fraction. In a reaction $A+B \rightleftharpoons C+D$, where the boiling sequence is A, C, D, B, this means that two additional distillation columns are needed in order to purify the products C and D. Nevertheless, in this case too there are often advantages over a reaction in a separate reactor, since the distillation column makes it possible to utilize the counter-current principle and functions under more advantageous concentration conditions.

In these cases, however, the important principle of utilizing counter-current flow is adversely affected by the overlap of reaction and separation by distillation; for example, in the last-mentioned case, increasing the heat input does not always result in an improvement in the conversion, since, as a result of the higher reflux ratio, the reactants and the catalyst in the reaction zone are diluted to a greater extent.

For example, in the case of the reaction $A+B \rightleftharpoons C+D$, where the boiling sequence is A, C, B, D and reactant A is introduced in excess, these effects, of which to date no account has been taken in the specialist literature, have the consequence that a particular result obtained in a distillation column in respect of conversion and take-off concentration is achieved for not only a fixed, pre-

determined heat input but also a substantially higher heat input. Two different operating states are possible. Simulation calculations have shown that the reason for this very surprising behavior is that in the case where the heat input is lower the counter-current flow can still be very effective, whereas in the other case it is weakened by excessive back-mixing. The energy input has to be increased several-fold in order to compensate for this deficiency by concentrating a reactant by distillation column are substantially lous. From the statements above, it can be seen that unrestricted overlap of reaction and distillation is not reasonable in every case.

It is an object of the present invention to employ a simple measure, which can be varied to suit a very large variety of distillation columns, to ensure that the distillation energy required for the separation of substances does not have an unnecessarily pronounced adverse effect on the counter-current flow of the reactants. Furthermore, it is intended to avoid the disadvantage arising from the need for additional separation columns for purifying the product streams when the reactants and the reaction products exhibit disadvantageous boiling behavior.

In accordance with the invention, it has been found that this object is achieved by a method of carrying out a chemical reaction and simultaneously separating a product mixture into several fractions by means of a distillation column which, in parts, is divided into a reaction section and a distillation section by separating units which are effective in the longitudinal direction and completely or partly prevent cross-mixing of liquid and vapor streams, wherein one or more reactants are fed into the reaction section, and at the same time one or more medium-boiling

fractions, which consist of reactants and reaction products and are free, or substantially free, from contamination by top and bottom fractions, are taken off in vapor or liquid form from the distillation section.

5

If desired, a catalyst may optionally be fed into the reaction section.

10 For the case of a simple distillation, the distillation column contain, in parts, separating units which are effective in the longitudinal direction and are intended to prevent cross-mixing of liquid and/or vapor streams. _____

A

An embodiment of the invention is shown in the drawing, and is described in detail below.

Figure 1 shows a flow diagram of a distillation column for the reaction $A+B \rightleftharpoons C+D$ (boiling sequence A, C, B, D; reactant A introduced in excess), where the product streams A, C and D are obtained by distillation.

Figure 2 shows a flow diagram of a distillation column for the reaction $A+B \rightleftharpoons C+D$ (boiling sequence A, C, D, B), where the product streams A, B, C and D are obtained by distillation.

In Figure 1, a column 1 is divided, above and below the feed points for the reactants A and B, into a reaction section 3 and a distillation section 4 by a separating unit 2 which is effective in the longitudinal direction. In accordance with the boiling sequence, the lowest boiling reactant A is taken off at the top, and the highest boiling reaction product D is taken off at the bottom of the column. The reaction is suppressed in the distillation section 4, and it is therefore possible to take off the medium-boiling product C in pure or substantially pure form from this section.

As shown in Figure 2, it is also possible with the same arrangement to use a single distillation column to achieve the envisaged object, ie. to obtain the reaction products C and D in pure form, for the particularly unfavorable case where the two products are neither the lowest boiling nor the highest boiling fraction and conventional procedures require the use of two additional distillation columns. By appropriately dividing the liquid and vapor streams between the reaction section and the distillation section, the counter-current principle is optimally effective and the overall expense can be reduced.

This longitudinal partition makes it possible to obtain a separate column section in which the reaction can take place under the optimum conditions in respect of heat consumption, back-mixing and catalyst concentration. The catalyst, which is generally non-volatile or of low

volatility (eg. mineral acids for esterifications, hydrolyses, acetal formation, acetal cleavage or transacetalization, or alcoholates for transesterifications) but may also be highly volatile (eg. hydrogen chloride or formic acid)

5 can be introduced into this part of the distillation column, ie. the reaction section, in a selective manner by appropriately choosing its feed point in accordance with its boiling behavior. Because the catalyst in the reaction section is less dilute, smaller amounts of catalyst

10 are generally required, or it is possible for the column to have simpler baffles which require a shorter residence time for the liquid, eg. packed columns instead of more expensive bubble tray columns or valve tray columns. This also has advantages when the residence time

15 is to be kept very short because of the thermal sensitivity of the products. The chemical reaction can be prevented in the distillation section opposite the reaction section by keeping the catalyst out of this area; this either results automatically because the catalyst is non-

20 volatile or of high volatility, or can be achieved in the distillation section by special measures, eg. deliberately destroying a catalytic acid by metering in an alkali, so that the only step taking place is the separation of substances by distillation. Sensitive substances which are

25 readily affected by the catalyst can be removed from this section in a mild manner. If desired, the reaction zone can be deliberately restricted even within the reaction section by, for example, limiting this zone to only the region between the feed points for the reactants.

30

There are several possible embodiments of the separating units which are effective in the longitudinal direction. The simplest of these is a flat, continuous separating sheet, but 2 concentric distillation columns or two such

35 columns arranged side by side can, if desired, also be used.

The chemical reaction may also take place in a

distillation column in which the residence time is increased by means of separate delay tanks which are located outside the distillation column and in which the reaction is, if required, catalyzed by an ion exchanger.

- 5 The longitudinal partition of the distillation column according to the present invention is particularly advantageous with respect to the purity of the products taken off.

The embodiments of the invention in which an exclusive property or privilege is claimed are defined as follows:

1. A method of carrying out a chemical reaction and simultaneously separating a product mixture into several fractions by means of a distillation column which, in parts, is divided into a reaction section and a distillation section by separating units which are effective in the longitudinal direction and completely or partly prevent cross-mixing of liquid and vapor streams, wherein one or more reactants are fed into the reaction section, and at the same time one or more medium-boiling fractions, which consist of reactants and reaction products and are free, or substantially free, from contamination by top and bottom fractions, are taken off in vapor or liquid form from the distillation section.

2. A method as claimed in claim 1, wherein a catalyst is also fed into the reaction section.

3. Apparatus for carrying out the method as claimed in claim 1 or 2, wherein the reaction section and the distillation section are located in a single distillation column and are separated from one another by separating units which are effective in the longitudinal direction.

4. Apparatus for carrying out the method as claimed in claim 1 or 2, wherein the reaction section and the distillation section, in part or over their entire length, are designed as two distillation columns separated from one another.



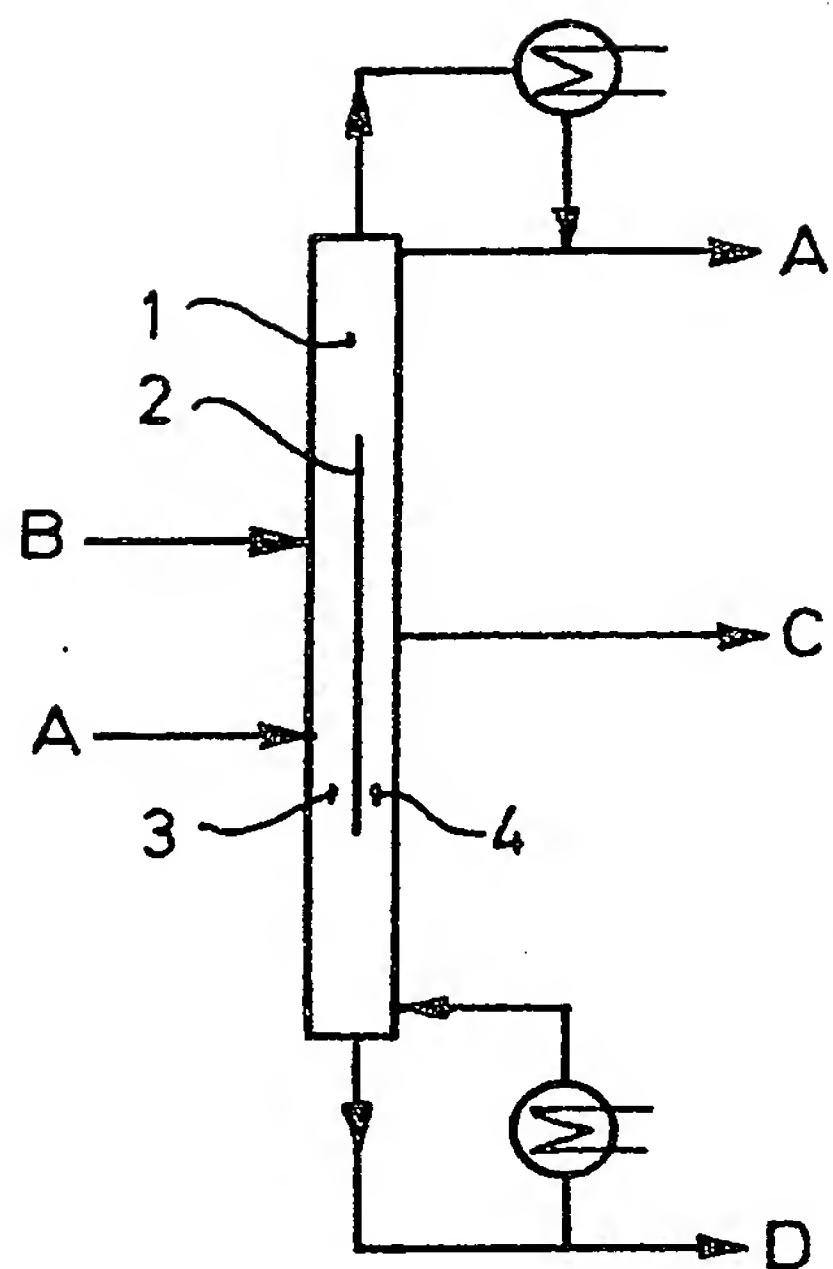


FIG.1

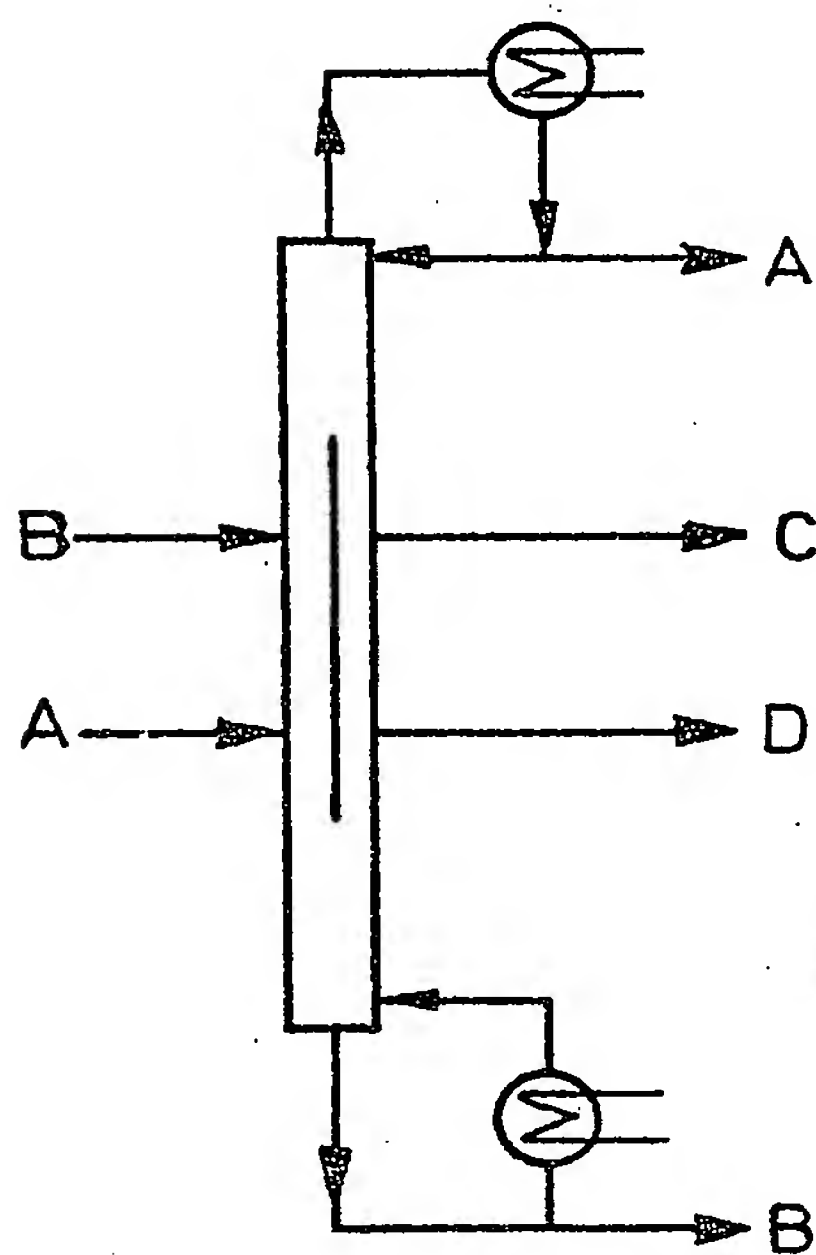


FIG.2

[Handwritten signature]
0050 / 36 479 Patent Agents

**This Page is Inserted by IFW Indexing and Scanning
Operations and is not part of the Official Record**

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

- ☒ **BLACK BORDERS**
- ☒ **IMAGE CUT OFF AT TOP, BOTTOM OR SIDES**
- ☒ **FADED TEXT OR DRAWING**
- ☐ **BLURRED OR ILLEGIBLE TEXT OR DRAWING**
- ☒ **SKEWED/SLANTED IMAGES**
- ☒ **COLOR OR BLACK AND WHITE PHOTOGRAPHS**
- ☒ **GRAY SCALE DOCUMENTS**
- ☐ **LINES OR MARKS ON ORIGINAL DOCUMENT**
- ☐ **REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY**
- ☐ **OTHER:** _____

IMAGES ARE BEST AVAILABLE COPY.

As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.